

- 2 -

Another destruction process, with recovery of certain components, is based on pyrolysis. The recycling consists in recovering the pyrolysis oil, the carbon and the metals. These processes are high-performance processes
5 but they require the investment of large sums of money and high treatment costs, they are often the source of significant atmospheric pollution.

Thus efforts have been intensified to develop methods for stripping this bulky waste in a manner which
10 is effective, economical and environmentally non-polluting. Considerable research has been focused on the problem, which obviously does not concern only worn tyres but all manufactured products made of rubber and also the waste accompanying the manufacture of these
15 products, these often being reinforced with metal reinforcements or reinforcements made of synthetic materials. The ultimate problem does not reside only in the economical destruction of the rubber element or of the tyres, but it is also desirable to be able to recover and
20 reuse them, or some of the components, for the purpose of avoiding unnecessary wastage of beneficial materials.

With the aim of illustrating the prior art in this field, it is appropriate to mention the following publications:

25 *Sub 3* - GB 2,026,144 (1979) of writing [sic] a plant for the treatment of rubber waste and of synthetic materials coming from worn tyres. Thermal decomposition of the product, which is coarsely reduced, is carried out in a fluidized bed of sand at 800°C in the presence of oxygen.
30 The gases produced by the decomposition are used and, at the end of the process, the reinforcing metals are recovered by means of magnets.

- US 4,426,459 (prior. JP 1980) discloses a process for the decomposition of vulcanized rubber by a treatment

- 3 -

around 100°C with an organic solvent in the presence of an alkaline hydroxide such as potassium hydroxide.

5 - DE 3,313,470 (1983) relates to a method and to an apparatus for treatment at a temperature above 200°C for 10 to 35 minutes in a liquid phase composed of spent mineral oils and of organic solvents. A viscous product is obtained which can be used as an additive in bitumens or insulation layers.

10 - WO 97/1561 (prior. US 1995 and 1996) describes a process for the devulcanization of rubber coming from downgraded tyres by desulphurization at around 300°C by means of an alkali metal in a medium that does not contain oxygen, before or during a treatment by an organic solvent after having separated the constituents of the tyres, such
15 as the steel and other reinforcements.

Many of these processes have disadvantages, namely from the standpoint of the investments, of the efficiency, of the complexity, of the environment or of the market.

ins
al
20 ay The object of the present invention is to remedy these drawbacks and to propose a process and a simple plant allowing separation of vulcanized and/or bonded rubber waste, such as tyres, belts or other objects, and allowing the constituents to be recovered for reuse by industry, without endangering the environment. The
25 separation of the rubbers from the other materials and metal or other reinforcements is easily accomplished, quickly and economically, the reinforcements made of synthetic resins being dissolved and the metal recovered. Thus, it is possible to provide industry with quality
30 products at competitive prices.

The process essentially comprises the following steps:

A) The reinforced or non-reinforced, vulcanized- rubber waste is coarsely reduced by cutting it up by means
35 of a saw, preferably of the guillotine type fitted

- 4 -

with cutters in a grating in order to obtain fragments 10 to 25 cm in length. The sole purpose of this is to reduce the size and thus facilitate handling during the treatment process.

- Sub a5
- B) The reduced waste is introduced into a reactor and treated for 30 minutes at 350°C with an OH⁻ ion generator, preferably a strong alkaline base such as molten NaOH. 3.[sic] Separation of the basic liquid and the residues coming from the treated rubber.
- 10 C) Neutralization of the residues with an acid, such as phosphoric acid;
- D) Recovery and separation of the rubber constituents and the metals used for reinforcement.

15 Destruction of some of the bonds between the rubber and the other reinforcing materials is achieved by the action of a strong alkaline base, such as molten NaOH which is maintained at a temperature of 350°C for approximately 30 minutes.

20 It is important to point out that the consumption of basic agent is very low and the etching liquid can be reused several times, by recovery and reinjection; the volume of the NaOH liquid product circulating will be automatically readjusted if necessary by a fresh addition of the product. Moreover, it should be emphasized that the

25 process according to the present invention does not involve any organic solvent. In addition, it works with waste which is only coarsely cut up and the substances serving for the proposed treatment are well known, commonly employed and inexpensive.

30 The plant for implementing the process is relatively simple and does not entail inordinate investment.

all

a6 The details of the process according to the present invention will emerge from the description of the

plant which relates thereto, the description being given by way of non-limiting example.

- Figure 1 shows schematically the overall plant;
- Figure 2 shows, on a larger scale, the first part of one of the possible embodiments of the plant;
- Figure 3 shows, on a larger scale, the second part of one of the possible embodiments of the plant.

As shown in Figure 1, crystallized NaOH in its original package is melted in the oven 1, at a temperature of 300 to 400°C, before being introduced into the master tank 5, which is provided with a heater and in which the NaOH is maintained at a temperature of 380°C before being transferred to the reactor 13 into which the waste coming from the cropper 14 is also introduced. After 30 minutes of immersion and with stirring at the start of obtaining a temperature of 350°C [sic]. The liquid is conducted by the line 19, furnishes of [sic] a pump, to the buffer tank 20 and then sent to the master tank 5. The buffer tank is heated to a temperature of 380°C in order to prevent heat shocks occurring in the tank 5. Moreover, the buffer tank is designed to gather the precipitates and is organized for the separation and extraction of the small particles. The decomposition products from the reactor 13 are transferred to the neutralization tank 23 and, at the end of the treatment, the residues are transported to the magnetic sorting device 32 where the metals are separated from the polymers resulting from the treatment.

According to this embodiment illustrated in greater detail in Figures 2 and 3, a drum 0 containing crystallized NaOH is introduced into the oven 1, which matches the shape of the drum, in which oven the NaOH is heated to a temperature of at least 380°C. The upper part and the straight vertical part of the oven form a cover which opens about the spindle 2 fastened to the bottom right of the oven in order to allow the drum to be easily

- 6 -

loaded. A connection tube running to the pump 3 is introduced and connected to the bung of the drum.

The liquified NaOH is transferred to the master tank 5 in which it is maintained at 350°C.

5 *50097* The master tank 5 is equipped with conventional monitoring and control instruments 8 to 11, known to those skilled in the art, which monitor the conditions in the tank and actuate the electronic control actuators when transfers are made and when other actions are taken. The
10 regulatory safety valve 7 prevents unexpected and accidental overpressures and the heating element 6 (may be placed [sic] on the outside, between the insulation, by heating coils conveying a liquid lime) keeps the temperature constant inside the tank.

15 The liquid is sent via the line 12, fitted with a pump, into the reactor 13 and the waste, cut up in the cropper 14, advantageously a guillotine, is sent by the chain conveyor 15 into the reactor 13, the solid materials must be immersed, the amount of caustic liquid is attained
20 by the operation and control of a probe which also actuates the closure of the valve 17 and the start of the stirring provided by the mixer 16. The temperature of the reactor is maintained by the heating system 18. After approximately 30 minutes treatment at 350°C, the materials
25 are deconsolidated, the caustic liquid is extracted by the line 19, fitted with a pump, through a filter 21, to the buffer tank 20. The filter 21 retains the particles greater than 1 mm. It is unclogged suddenly by closing the valve of the line 19 and sending compressed air 44 into
30 that part of the line 18 [sic] which is connected to the tank 13.

The buffer tank 20 is equipped with the same monitoring, control and heating apparatuses as the tank 5; it has a configuration which allows settling and reheating
35 of the caustic liquid to the temperature of 350°C in order

to avoid thermal shocks and the risk of crystallization in the master tank 5. The liquid in the decanted part and at the correct temperature in the buffer tank 20 is then sent back via the line 21, by gravity, slowly into the master tank 5.

When all the caustic liquid has been withdrawn from the reactor 13, the large valve 22 is opened and releases, by gravity, the materials in the reactor 13 into the neutralizing tank 23.

10 The neutralizing liquids for the above treatments
and for cleaning the plant, coming from the tank 24, are
conveyed by the line 25, fitted with a pump, to the
neutralizing tank 23 into which the liquid is injected by
sprayheads. The phosphoric acid coming from the tank 26
15 passes via the mixing unit 27 through the line 25. The
materials and the liquid are vigorously stirred. The pH
meter 11 sets the $pH = 7$ after a little more stirring,
there is a hold period in order to allow the suspended
materials to precipitate, the neutralized liquid is
2. extracted just at the point of connection of the line 28,
fitted with a pump, to the tank 23. Injections of air 44
into this line have the purpose of clogging the filter
installed at the end of the line 28.

The neutralized liquid having been withdrawn, the large valve 29 is opened while the valve 30 remains closed. The neutralized liquid is found in the tube with the materials between the two valves 29 and 30, it is withdrawn through the bypass of the line 28, the bypass also being equipped at the end of the line with a filter unclogged by sudden injections of air 44, and then the liquid is directed into the tank 24. After removing the rest of the liquid, the valve 30 is gradually opened so as to release the solid material coming from the neutralizing tank 23.

- 8 -

The material is transported by a conveyor belt to the magnetic pulley 32 located at the end of the belt. The metals 33 are precipitated into the container 34 and the non-magnetic materials 35 end up in the container 36. The container 36 is provided with a double bottom, the material being deposited on the first bottom provided with a stainless steel filter whose porosity is less than 10 microns. The material is dried by liquid flow via a lateral pipe 45. The containers 34 and 36 are superposable and have opening bottoms.

Liquified NaOH has a boiling point greater than 1000°C; there is no pressure formation in the plant, however the equipment is designed to withstand a pressure of 10 bar in anticipation of possible heat shocks.

15 Sub 2107 As this is a heat treatment, it is preferable to
work continuously as long as possible. For this purpose,
and in order to avoid shut downs due to the build-up of
impurities and small particles, cleaning devices are
provided for removing them, without having to stop the
20 production. After a number of treatments and in
anticipation of cleaning the buffer tank 20, the NaOH
liquid in the master tank 5 will be taken to the minimum
level in order to receive the entire solution stored in
the buffer tank 20, up to the level of the tap on the line
25 21. Next, water will be slowly added via the line 37 to
the rest of NaOH liquid, bottom of the buffer tank 20 for
a direct dilution to the point of non-crystallization
(concentration +/- 40%). After this dilution, the valve 38
is opened on a vibrating screen 39 having a porosity of 10
30 microns. The solid particles are removed to a container 40
and will be able to be transferred thereafter to a washing
tank external to the system with a filter press for the
impurities precipitated by the neutralization and for
recovering the materials to be recycled in a containers
35 [sic] of the 36 type. The 40% NaOH solution is recovered

~~outlet 41
use or e
ain a neu
bility of~~

10

15

20
~~Suball~~ [sic]
possib
shut-o
buffer

30

neutralization centre for these acids, constituting an appreciable financial plus, thus reducing the cost of the main treatment forming the subject of the present invention.

5 The recovered metals will be sent to the steel industry.

10 The other materials recovered from the tyres are friable and are converted into a fine powder with slight pressure. Devulcanization is not complete but the breaking of certain bonds is ensured. The friable nature and the polymeric composition of the residue make thereof a beneficial filler material, to be recycled in the manufacture of tyres and rubber articles, and for other non-limiting applications, such as in bitumens or other
15 bitumenous mixes.

 It is obvious that the present invention is in no way limited to the embodiment as described in the illustrative embodiment shown in Figures 1, 2 and 3. Variants may be made to it without thereby departing from
20 the scope of the claims.